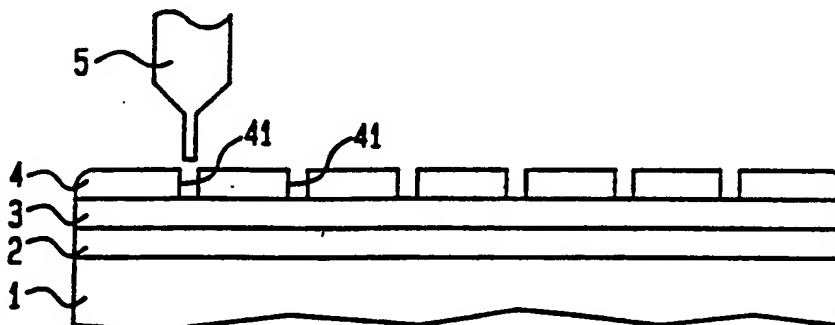


## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(21) International Application Number:</b> PCT/US91/00729 <b>(22) International Filing Date:</b> 4 February 1991 (04.02.91) <b>(30) Priority data:</b> 516,173 30 April 1990 (30.04.90) US <b>(71) Applicant:</b> BELL COMMUNICATIONS RESEARCH, INC. [US/US]; 290 West Mount Pleasant Avenue, Livingston, NJ 07039-2729 (US). <b>(72) Inventors:</b> GMITTER, Thomas, John ; 448 Lonna Court, Lakewood, NJ 08701 (US). YABLONOVITCH, Eli ; 11 Blueberry Lane, Leonardo, NJ 07737 (US). <b>(74) Agents:</b> WINTER, Richard, C.; PCT International, Inc., Post Office Box 573, New Vernon, NJ 07976 (US) et al.		<b>(81) Designated States:</b> AT (European patent), BE (European patent), CH (European patent), DE (European patent), DK (European patent), ES (European patent), FR (European patent), GB (European patent), GR (European patent), IT (European patent), JP, LU (European patent), NL (European patent), SE (European patent).  <b>Published</b> <i>With international search report.</i>

**(54) Title:** PATTERNING METHOD FOR EPITAXIAL LIFT-OFF PROCESSING**(57) Abstract**

In the fabrication of microelectronic, optoelectronic, and photonic devices, methods are being used which involve selective processing of material in the presence of a patterned mask layer; for example, such processing may involve etching. Typically, mask layer material is chosen on the basis of response to suitable radiation, allowing for patterning by selective irradiation followed by selective removal of mask layer material. However, in so-called epitaxial lift-off processing, material (2, 3) to be processed may be covered with a support layer (4) of a material which is selected in view of desired mechanical and thermal properties, and which is not amenable to patterning by radiation. A method is described which provides for patterning of such layer by heated mechanical means (5, 9) such as, e.g., a heated stylus or roller.

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PATTERNING METHOD FOR EPITAXIAL LIFT-OFF PROCESSING

This invention was made partially with Government support under Contract DAAL01-89-C-0900 awarded by the Department of the Army. The Government has certain rights in  
5 this invention.

Technical Field

The invention is concerned with pattern formation in the fabrication of microelectronic, optoelectronic, and photonic devices.

10 Background of the Invention

The fabrication of microelectronic, optoelectronic, and photonic devices typically involves numerous steps, including layer formation, dopant implantation, and selective pattern processing-- resulting in a desired structure to be  
15 electrically contacted and/or optically accessed. In this context, special attention will be paid in the following to pattern processing as applicable, e.g., in pattern etching, and especially to the patterning of a mask layer prior to processing of underlying material.

20 On a material to be processed, e.g., by etching, a mask or "resist" layer may be formed of a material which is sensitive to suitable radiation such as, e.g., visible or ultraviolet light, electrons, x-rays, or ions. After such a material has been selectively exposed to radiation, exposed or  
25 unexposed areas may be selectively removed by a chemical agent which leaves complementary areas essentially unaffected.

In the practice of such methods, the choice of mask material is preferably based on radiation response. However, there are instances in which material to be processed is  
30 coated with an auxiliary layer whose material composition is selected on the basis of different criteria: for example, in so-called epitaxial lift-off processing of an epitaxially grown layer, a support layer material may be chosen mainly on

account of desirable properties including a low Young's modulus, a high coefficient of expansion, high adhesion, and high chemical durability.

Epitaxial lift-off processing involves growth of a  
5 desired film on a first, auxiliary or growth substrate,  
followed by removal of such film onto a second, desired device  
substrate. Such processing is receiving attention, e.g., as  
an alternative to lattice-mismatched epitaxial growth, the  
combination of silicon and Group III-V technologies currently  
10 being of particular interest. For example, as disclosed by E.  
Yablonovitch et al., "Extreme Selectivity in the Lift-off of  
Epitaxial GaAs Films", Applied Physics Letters 51 (1987), pp.  
2222-2224, a gallium arsenide layer can be grown on an  
intermediary aluminum arsenide layer on a gallium arsenide  
15 substrate, and the grown gallium arsenide layer can be lifted  
off upon undercut etching-- i.e., upon chemical dissolution of  
the intermediary layer.

Epitaxial lift-off is facilitated by use of a support  
layer which is formed on the layer to be lifted off, and it  
20 was found that a material known as Apiezon-W is particularly  
suited for this purpose. Indeed, use of such material results  
in preferred edge-curling during undercut-etching; see U. S.  
patents 4,846,931 and 4,883,561, issued to T. J. Gmitter et  
al. on July 11, 1989 and November 28, 1989, respectively. For  
25 exemplary uses of resulting films as bonded to desired  
substrates see, e.g., E. Yablonovitch et al., "Regrowth of  
GaAs Quantum Wells on GaAs Liftoff Films 'Van der Waals  
Bonded' to Silicon Substrates", Electronics Letters, Vol. 25  
(1989), p. 171; and E. Yablonovitch et al., "Double  
30 Heterostructure GaAs/AlGaAs Thin Film Diode Lasers on Glass  
Substrates", IEEE Photonics Technology Letters, Vol. 1 (1989),  
pp. 41-42.

Further with respect to epitaxial lift-off processing,  
as single-crystal layers typically are incorporated in devices  
35 or "chips" which are considerably smaller than a growth  
substrate or "wafer", and since lift-off of epitaxially grown

layers is facilitated when the size of such layers does not exceed a practical limit, it is desirable to provide a means for partitioning a grown semiconductor layer prior to epitaxial lift-off. However, as an instance of a difficulty  
5 mentioned above, such partitioning is impeded in the presence of a support layer which is not amenable to patterning by selective irradiation. The invention described below is motivated by the desire for a patterning method which is applicable in this situation, and which does not rely on the  
10 use of radiation.

### Summary of the Invention

In epitaxial lift-off processing, patterning of a support layer on a structure comprising an epitaxially grown layer on a substrate is effected by heated mechanical means  
15 such as, e.g., a heated stylus or roller. Underlying material is partitioned by etching through grooves or channels formed by such patterning, and undercut etching is used to detach partitions from the substrate.

### Brief Description of the Drawing

20 FIG. 1-4 are schematic elevations of a structure at sequential stages of processing in accordance with a preferred embodiment of the invention in epitaxial lift-off processing;

FIG. 5 is a schematic perspective view of exemplary patterning apparatus comprising a stylus in accordance with a  
25 preferred embodiment of the invention;

FIG. 6 is a schematic perspective view of an assembly comprising a plurality of stylus as may be preferred in accordance with an alternate embodiment;

FIG. 7 is a schematic side view of an assembly  
30 comprising a heated roller and a wiper or scraper as may be used in accordance a further preferred embodiment of the invention; and

FIG. 8 is a schematic frontal view of an assembly comprising a roller provided with a plurality of rims as also

may be preferred.

For the sake of ease of appreciation structural dimensions are not drawn to scale in the Figures.

#### Detailed Description

5 In the Figures, like features are identified by the same numerals.

FIG. 1 shows an assembly 10 comprising growth substrate 1 (consisting, e.g., of doped or undoped gallium arsenide), epitaxial release layer 2 (consisting, e.g., of aluminum  
10 arsenide and having a thickness in a typical range from 2 to 1000 nanometers), epitaxial layer 3 (consisting, e.g., of doped or undoped aluminum gallium arsenide)-- to be lifted off for removal to a different substrate, and support layer 4 (consisting, e.g., of "black wax" paraffinic-asphaltene, a  
15 wax-like material also known by the trade name Apiezon-W.) Layer 4 may have a thickness in a typical range from 10 to 1000 micrometers, and its formation may be by melting-on, or by painting or spraying in solution; in the case of Apiezon-W, trichloroethylene is suitable as a solvent. Also, formation  
20 of layer 4 may include heating (to a temperature of approximately 160 degrees C in the case of Apiezon-W), followed by quenching, such processing being beneficial in the interest of increased stress at the interface between an epitaxial layer and its support layer. This, in turn, can  
25 result in advantageous edge-curling in a later removal step as described below in connection with FIG. 4. Support layer 4 may be provided with mechanical implants by which later removal is facilitated.

Suitability of a layer-4 material with respect to  
30 preferred processing of the invention is readily ascertainable by testing a layer by means of a heated stylus or roller, preferred temperature being chosen as a function of speed of motion, with higher temperatures corresponding to higher speeds and conversely. Preferred speeds also depend directly  
35 on stylus or roller pressure which, in the interest of

minimizing mechanical damage to layer-3 material, may be controlled by spring loading at an essentially constant low level.

While, for the sake of clarity and ease of exposition, layer 3 is shown without structural features, it is understood that such layer may actually comprise a number of sub-layers which may be patterned and doped as, e.g., in the fabrication of microelectronic devices. This may involve inclusion of dielectric as well as of metallic layers which, typically, have amorphous or polycrystalline rather than epitaxial structure.

FIG. 2 further shows stylus 5, having been used on the structure of FIG. 1 to produce grooves or channels 41 in layer 4 by scoring; alternatively, grooves 41 can be produced by a roller. Such stylus or roller may be made, e.g., of stainless steel, preferably Teflon-coated, or of Teflon sandwiched between stainless steel support members. Typical width of the tip of a stylus or the edge of a roller is in the range from 5 to 100 micrometers, and, in the case of Apiezon-W, preferred stylus or roller temperatures are in a range from 100 to 200 degrees C, while essentially no pressure is applied. Typical stylus or roller speed is approximately 3 centimeters per second.

FIG. 3 shows the structure of FIG. 2 after (selective) etching of layer 3 via grooves 41, the patterned support layer 4 having served as a mask. In the case of gallium arsenide such etching may be carried out, e.g., by means of a mixture comprising 1 part sulfuric acid, 8 parts hydrogen peroxide, and 500 parts water by volume. Similarly suitable is a mixture of ammonium hydroxide and hydrogen peroxide.

FIG. 4 shows the structure of FIG. 3 after undercut-etching of lift-off layer 2. In the case of aluminum arsenide such etching may be effected, e.g., by means of 10-percent hydrofluoric acid at a temperature near 0 degrees C. (This etchant was found to exhibit very high selectivity with respect to etching of aluminum arsenide while leaving

certain aluminum gallium arsenide alloys essentially intact.) As mentioned above, in the presence of suitable strain in the structure to be lifted off (such strain being introduced by support layer 4), undercut-etching is facilitated by edge-curling, allowing reaction products such as hydrogen gas to escape, thereby permitting more complete penetration of the etchant between substrate 1 and layer 3. At this point, supported several films 31 can be picked up (mechanically or pneumatically, for example) or floated off for placement in an intended device environment. After such placement, layer-4 material may be removed by means of a chlorinated solvent.

FIG. 5 illustrates apparatus by which preferred processing may be carried out, including substrate support 6 on x-y stage 7, stylus 5 attached to stylus support 8 which is equipped with electrical resistance heater 9.

FIG. 6 illustrates a detail variation of apparatus of FIG. 5, comprising a plurality of styluses in a rake-like structure.

FIG. 7 shows a further variation of apparatus of FIG. 5, comprising roller 11 with rim 111, holder 12, support 8 with heater 9, (optional) wiper or scraper 13, and base 14 to which support 8 and wiper 13 are attached. Use of one or several rollers may be preferred over the use of styluses in the interest of precise corners in the case of intersecting grooves. A plurality of rollers may be used analogous to a plurality of styluses; also, as illustrated by FIG. 8, a roller 11 may be provided with a plurality of rims 111.

Grooves or channels may be formed by back and forth motion of a stylus or roller, or by motion of a stage on which the substrate is placed. Motion may be rectilinear or curvilinear, and may be controlled, e.g., by mechanical track following, or by computer commands. Commercially available stages, transits, or transports may be used.

Preferred processing in accordance with the invention is similarly applicable in epitaxial lift-off processing of structures grown on other types of substrates such as, e.g.,



Claims

1. In the fabrication of microelectronic, optoelectronic, or photonic devices, a method for removing a structure comprising an epitaxial film from a substrate, comprising the steps of
  - 5 forming on said structure a layer of a support material,
  - patterning said layer of support material by heated mechanical means, thereby selectively exposing material underlying said layer of support material,
  - 10 etching exposed underlying material, and
  - undercut-etching of material underlying said structure.
2. The method of claim 1, said support material comprising paraffinic-asphaltene wax.
3. The method of claim 1, said support material having a thickness in the range from 10 to 1000 micrometers.
4. The method of claim 1, said patterning producing a channel having a width in the range from 5 to 100 micrometers.
5. The method of claim 1, said heated mechanical means comprising a stylus.
6. The method of claim 5, said heated mechanical means comprising a plurality of styluses.
7. The method of claim 1, said heated mechanical means comprising a roller.
8. The method of claim 7, said heated mechanical means comprising a plurality of rollers.
9. The method of claim 7, said roller comprising a plurality of rims.

-9-

10. The method of claim 7, further comprising wiping of a channel formed by said heated mechanical means.

11. The method of claim 7, further comprising scraping of a channel formed by said heated mechanical means.

12. The method of claim 1, patterning resulting upon rectilinear relative motion between said substrate and said heated mechanical means.

13. The method of claim 1, patterning resulting upon curvilinear relative motion between said substrate and said heated mechanical means.

14. In epitaxial lift-off processing, a method for selectively freeing an epitaxial film from a substrate upon which it was grown, comprising the steps of

forming on said film a support layer,  
5 patterning said support layer by heated mechanical means to selectively expose said film under said support layer, and  
etching said exposed film through said patterned support layer.

15. The method of claim 14 wherein said etching step includes undercut-etching of curled edges of said film.

FIG. 1

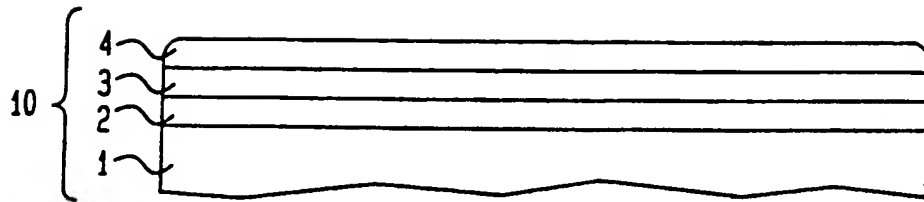


FIG. 2

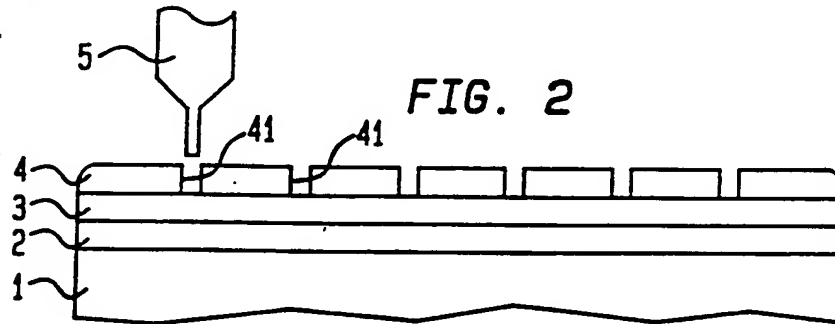


FIG. 3

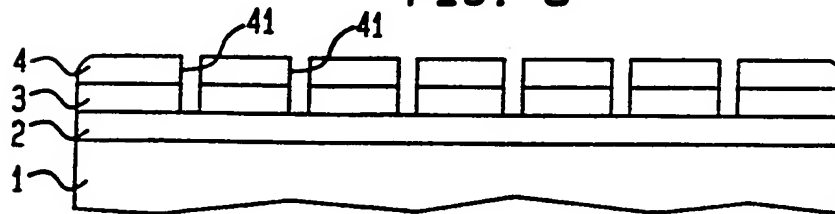


FIG. 4

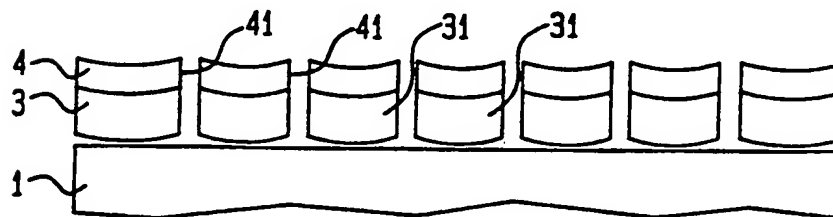


FIG. 5

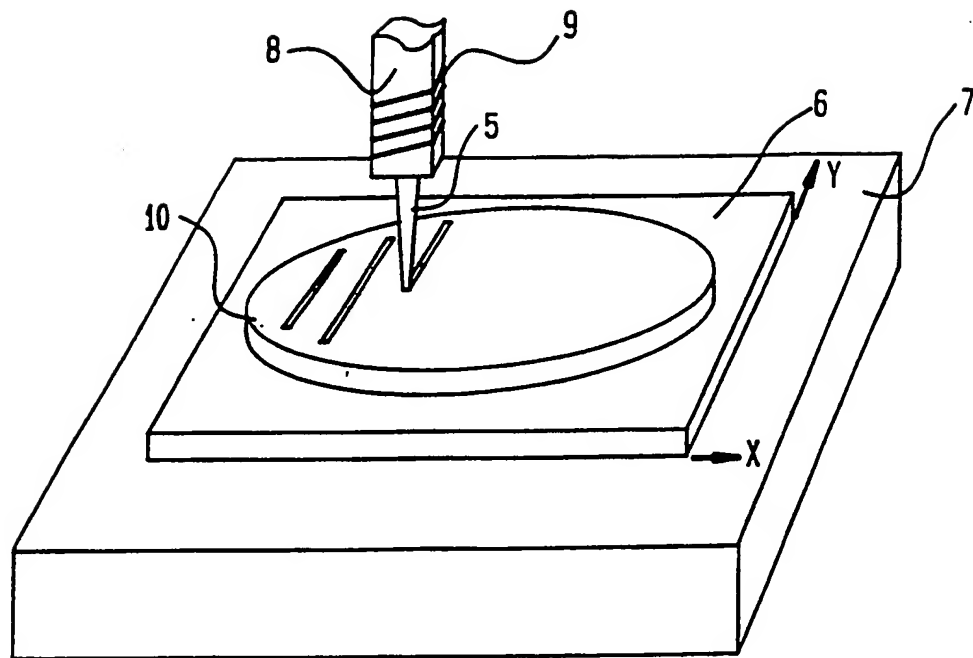


FIG. 6

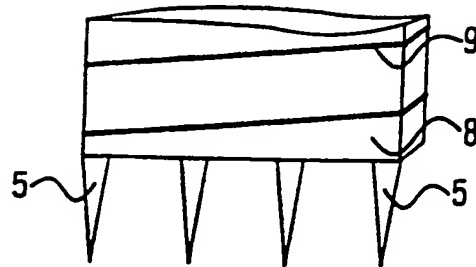


FIG. 7

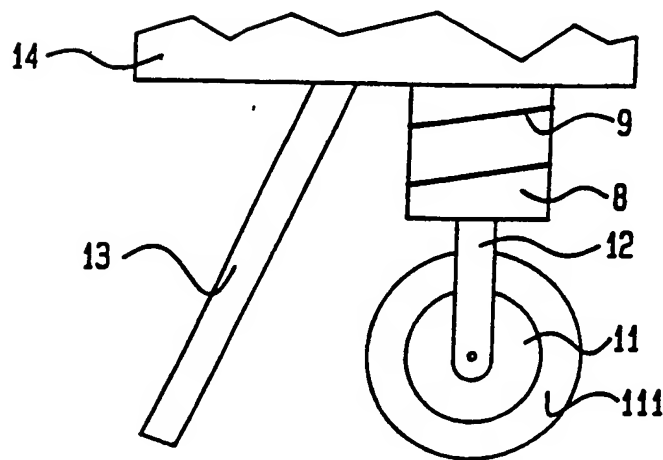
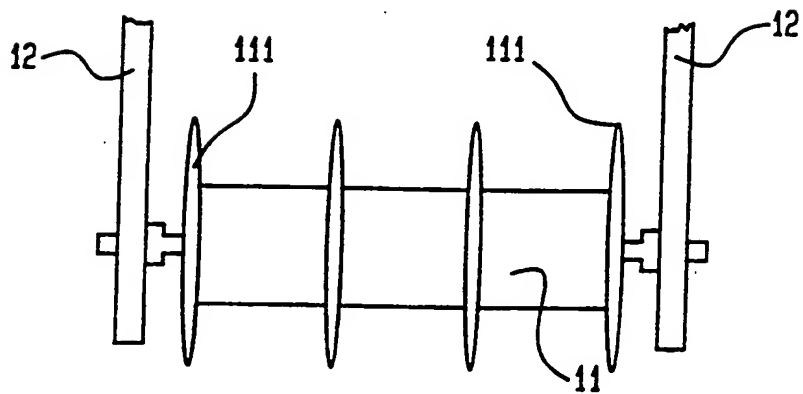


FIG. 8



# INTERNATIONAL SEARCH REPORT

International Application No **PCT/US91/00729**

## I. CLASSIFICATION OF SUBJECT MATTER (if several classification symbols apply, indicate all) <sup>3</sup>

According to International Patent Classification (IPC) or to both National Classification and IPC

IPC(5): H01L 21/00

US CL : 156/655,658,659.1,662

## II. FIELDS SEARCHED

Minimum Documentation Searched <sup>4</sup>

Classification System

Classification Symbols

US

156/632,645,655,658,662,659.1  
437/228

Documentation Searched other than Minimum Documentation  
to the Extent that such Documents are Included in the Fields Searched <sup>5</sup>

## III. DOCUMENTS CONSIDERED TO BE RELEVANT <sup>14</sup>

Category <sup>*</sup>	Citation of Document, <sup>16</sup> with indication, where appropriate, of the relevant passages <sup>17</sup>	Relevant to Claim No. <sup>18</sup>
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A	US, A, 3,290,192 (KELLEY) 06 DECEMBER 1966 See entire document.	1-15
A	US, A, 4,035,226 (FARBER ET AL.) 12 JULY 1977 See the entire document.	1-15
A	US, A, 4,325,779 (ROSSETTI) 20 APRIL 1982 See the entire document.	1-15
A	US, A, 4,818,336 (ROSSETTI) 04 APRIL 1989 See the entire document.	1-15
A	US, A, 4,883,561 (GMITTER ET AL.) 28 NOVEMBER 1989 See the entire document.	1-15
A	US, A, 4,846,931 (GMITTER ET AL.) 11 JULY 1989 See the entire document.	1-15

<sup>\*</sup> Special categories of cited documents: <sup>15</sup>

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## IV. CERTIFICATION

Date of the Actual Completion of the International Search <sup>2</sup>

25 APRIL 1991

Date of Mailing of this International Search Report <sup>2</sup>

**23 MAY 1991**

International Searching Authority <sup>1</sup>

ISA/US

Signature of Authorized Officer <sup>19</sup>

THI DANG